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The Wide Field/Planetary Camera 2 (WFPC-2) Molecular Adsorber

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Foreword

The intent of the authors of this report was to provide a complete record of the development of the Wide Field/Planetary Camera 2 molecular adsorber in order to establish priority for this novel accomplishment and to ensure that future work could build on a good foundation. Thus, I have chosen to rather extensively document even the minor steps and missteps taken in the process of moving from a good idea to flight hardware. I hope that some of the authors and the many others who made invaluable contributions in their own fields of expertise will also publish their work. Zeolite coatings promise a broad range of applications to molecular contamination control for spacecraft and instruments.

Jack Barengoltz
Cognizant Engineer



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Section 1

Background

The development, design, fabrication, and testing of a molecular adsorber for Wide Field/Planetary Camera 2 (WFPC-2) was part of a program to replace the WFPC-1 with a better instrument. In addition to the well-known objective for WFPC-2, the optical correction for the Hubble Space Telescope (HST) spherical aberration, there is a desire for imaging capability in the far ultraviolet (UV). The principal limitation to observations in the far UV is the condensation of contaminant gases on the cooled window of the light imaging array, a charge-coupled device (CCD). The molecular adsorber was an important component of the effort to significantly reduce the collection of molecular contamination on the CCD.

An ambitious specific contamination control goal (less than one percent relative loss in the response at 1470 Å due to the accumulation of molecular contamination in 30 days) was established in response to the needs of the scientists.^{1,2} This performance goal represents an estimated reduction in the molecular contaminant flux collecting on the CCDs of four to five orders of magnitude, compared to WFPC-1. For comparison, in system thermal vacuum testing, WFPC-1 typically lost 50 percent of its throughput at 1470 Å within hours of CCD cool down.¹ This flux arises from outgassing from the internal subsystems and materials. In flight, there is also a sizable contribution from the HST internal environment, especially for WFPC-2, with an open aperture for better UV performance and, on balance, less contamination on the CCD. (WFPC-1 has an aperture window.)

The original approach¹ included redesigns of WFPC-1 to provide "molecular baffles" and additional venting of the housing and a comprehensive program of in-process (i.e., prior to assembly) vacuum bakeouts of all internal and selected external instrument subsystems. "Molecular baffles" and housing vents were studied with the use of the Contamination Analysis Program (CAP) relative to the predicted internal sources.³ Only the open aperture, housing vents, and the venting of outboard electronics bays showed significant promise for the reduction of contamination accumulation on the CCDs. Therefore, Millard⁴ extended the original 5-in² housing vent area in a parametric CAP analysis up to 40 in². However, later "hand" calculations confirmed our early fears that venting to the HST environment, especially the housing to the HST aft shroud volume, would let in more contamination than would be removed. Thus, housing vents could not be used. (The outboard bay venting and open aperture were useful and implemented.) The data from the vacuum bakeouts, well underway in the summer of 1991, indicated that the best result to be expected in a reasonable duration (based on facility costs) and a reasonable temperature (based on hardware limitations) was an outgassing reduction by a factor of 100.⁵ Between the elimination of housing vents and the practical limitations of bakeout, WFPC-2 was going to miss its goal by about a factor of 100!

Therefore, in the summer of 1991, other options to achieve the goal were considered and analyzed for their potential advantage.⁵ Two options were selected by the project for implementation: a change to CCDs operable at warmer temperatures (for other performance advantages also), and the incorporation of molecular adsorbers inside the housing. The prediction for an increase in CCD operating temperatures from -100°C to -50°C was a factor of 20 reduction in the CCD accumulation in 30 days, based on source rate⁶ and bakeout data. Finally, a set of ideal molecular adsorbers with a total presented area of 40 in^2 was expected to reduce the accumulation by a factor of at least 10. This prediction was based on Millard's CAP analysis^{3,4} for housing vents. An ideal molecular adsorber in this application may be considered a one-way virtual vent for internal contaminants. An area of 100 in^2 was selected to provide a design margin. The activity to develop a WFPC-2 molecular adsorber was begun.

Section 2

Approach

2.1 Functional Parameters

The performance specifications of a molecular adsorber crucial to WFPC-2 are the capacity of the adsorbent for the contaminant molecules to be removed and the adsorbent's effective sticking coefficient for those molecules. In addition to the typical requirements of any WFPC-2 assembly, the molecular adsorber is required not to shed particles inside the instrument. Other issues include the outgassing in flight of water adsorbed during ground handling and the retention of the adsorbed contamination in vacuum at elevated temperatures.

The capacity of the adsorbent is a straightforward concept. Based on preliminary estimates of the relatively small amounts of contamination expected, the capacity was thought to be a potential sizing parameter for the final design and merely a valid selection criterion for the preliminary design. For the present discussion, the effective sticking coefficient is defined as the fraction of molecules incident on the adsorber that are adsorbed. In the intended use of the molecular adsorber as a one-way virtual vent for the instrument housing, its sticking coefficient is an exact sizing parameter. If the required area of real vent (coefficient of one) is known, then the required area of molecular adsorber is the product of the reciprocal of its sticking coefficient and the vent area. Thus, the sticking coefficient is both a preliminary and a final design factor. The particle shedding issue is related to the rather brittle nature of many adsorbents and the launch vibration environment. For the purposes of this work, shedding is meant to include the release both of pre-existing particles and of new particles generated by any mechanism. The other parameters were postponed to consideration in later phases of the development, based on theoretical and experiential grounds.

At the start of the development of the molecular adsorber, the capability of molecular adsorbents in general was not in question. The capacity was used as a simple adsorbent selection criterion. The sticking coefficient was estimated by various means to establish proof of concept for the selected adsorbent. The particle shedding issue, which was expected to be the greatest challenge, became the driver in design matters such as the physical form of the adsorbent, the preparation of the adsorbent, and the packaging of the devices.

2.2 Adsorbent

The adsorbent candidates were selected principally by their adsorption characteristics and capacities (extensive information in the literature), their

availability, and expediency. Although the contaminants of interest are unknown, certain judgements were possible. Simple alumina and silica, for example, were not chosen for consideration. There were two reasons for this: (1) these materials depend upon surface area that is principally a function of pore sizes that are physically controlled by the preparation process, and typically retain molecules by physisorption, rather than chemisorption, and (2) they are not highly selective for adsorbing organic compounds, particularly compounds which do not have high dipole moments, i.e., alkanes or alkenes, from the gas phase. Furthermore, since adsorption by these adsorbents is typically due to diffusion into the pores, the sticking coefficient (and rate of uptake) would undoubtedly be low. However, either alumina or silica can serve quite well as a binder to hold other materials that may possess more acceptable adsorptive behavior.

Charcoal is an excellent adsorbent for capturing organic compounds from either liquids or gases. However, the structural integrity of charcoal is poor at best. In commercial practice composite particle beds, not supported material, are typically used, and vibrational environments are uncommon. Carbon molecular sieves have a very high surface area, often exceeding 1000 m²/g. The pore sizes are commonly small, which limits the capacity for large molecule retention, since most of the high molecular weight organic compounds would be adsorbed on the external surface of the particles. At the Jet Propulsion Laboratory (JPL), solid monolithic carbon sorbents have been made, which could be utilized, if the carbon pore sizes permitted enough penetration of sorbate into the inner pores and cavities to provide adequate capacity. Carbosieve S, which can be produced in this form, was therefore included.

Zeolites^{7,8,9} are also very good adsorbents, with high surface areas, typically more than 200 m²/g. Zeolites are open framework, crystalline lattices, consisting of silica-aluminate or silica "cages" (Figure 2-1). The lattice of each type of zeolite has specific, controlled physical parameters. This feature offers significant advantages: the sorbent characteristics depend on the crystalline framework, which is uniform throughout the zeolite; the selectivity for and retention of sorbates by size is unique to specific, known crystalline dimensions; and the properties of the various sites within the lattice confer selective adsorption for different functional groups of organic molecules. The "cages" provide volumes with high available surface area, where multiple layers of sorbate may be adsorbed. The pore size and channel connections (between the "cages") of the frameworks determine the transport dimensionality and adsorption rates unique to different zeolite types.

The four zeolites chosen as candidates have differences that, to a first approximation, offer the potential for deciding on preferred sorption and retention properties for the particular class of compounds expected to be found in WFPC-2. Although all can be modified to take on different characteristics (e.g., cation exchange, aluminum content in the framework, hydrophobicity, etc.), other than those evaluated, the schedule and focus did not allow for this type of research-level investigation. Basically, the differences are structural and are as follows: Linde 4A has channel openings between large cells with 4.1 Å cross sections, Linde L has 7.1

Å channel openings, faujasite (Linde 13 X) has 7.4 Å channel openings, mordenite has two opening sizes, 6.5 X 7.0 Å in one direction and in another direction, 2.6 X 5.7 Å. Furthermore, Linde 4A has 3-dimensional access through the lattice, Linde L has 1-dimensional access, faujasite has 3-dimensional access, and mordenite has 2-dimensional, nonequivalent access. While these dimensional constraints do not necessarily add to the capacity value from a volume aspect, the ease of access into the lattice, to the more attractive sorbing sites, and the ability for the sorbates to form fit to the lattice framework would be expected to be important to the subsequent retention after adsorption.

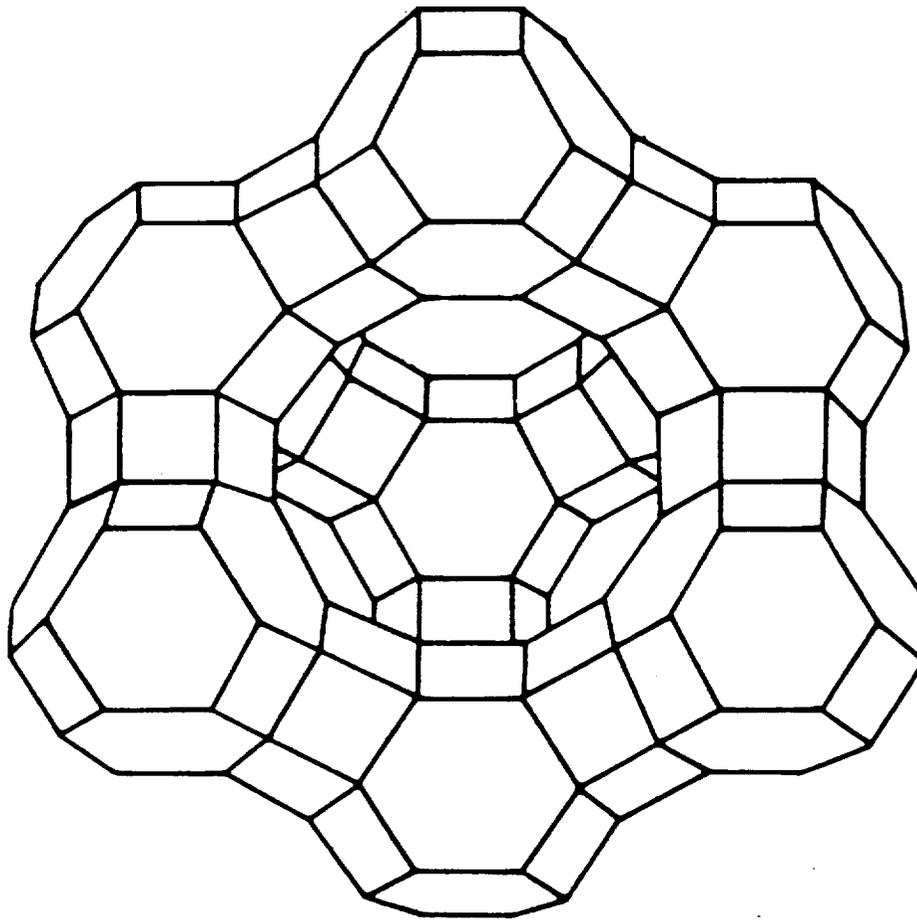


Figure 2-1. Structural model of faujasite (Y zeolite).

2.3 Substrate

Possible approaches to the packaging of the adsorbent in a flight configuration may be grouped into the categories of physical containment and coatings on a substrate. Physical containment is extremely simple, but presents the problem of sufficient apertures for the contaminant molecules to enter and be adsorbed without allowing particles of adsorbent to escape. Any access limitation proportionately reduces the effective sticking coefficient. In addition, a rigid container (e.g., wire mesh) might create a large quantity of fines through abrasion on a significant fraction of the adsorber surface area during launch. A compliant container ("zeolite beads in a bag") was considered briefly.¹⁰ However, the opening for the contaminant molecules and self-abrasion of the beads remain as problems, and additionally the bag material must not outgas. Therefore the development proceeded in the direction of adsorbent coatings on a substrate for zeolite (and also the monolithic carbon adsorber).

The substrate offering the greatest advantage is a monolithic material that has high surface area upon which sorbate molecules can be bound and which can be mounted at a number of different locations sufficiently well that it will not break or cause problems during any spacecraft operations. In order to enhance available surface area for effective adsorption of contaminants, the open-faced honeycomb monolithic structure was chosen. An illustration of this structure is given in Figure 2-2. This design offers the obvious advantage of a geometrical increase in the effective sticking coefficient of the adsorber over the sticking coefficient of the adsorbent on a flat substrate. This effect is exactly analogous to the increased effective infrared emittance (or by Kirchoff's law, the absorptance) of a coated radiator with surface wells over the emissivity (absorptivity) of the coating. Specifically, the probability of the adsorption of a contaminant molecule incident on the honeycomb face and into a cell is enhanced by the number of collisions with the adsorbent coating that the molecule might make before escaping the cell even if it were not adsorbed on the first collision. For a favorable geometry, an adsorbent with a sticking coefficient as little as 0.5 can provide an adsorber with an effective sticking coefficient of 0.95.

Various materials and channel densities were potential candidates for the coated substrate, but cordierite honeycomb monolith (Corning 9475), with a channel density of 400 cells per square inch was chosen because it: (1) is inert, (2) has no thermal stability problems under spacecraft conditions, (3) has low density, (4) will bind various sorbent materials to its surface and (5) is available in a variety of sizes¹¹ at a nominal cost. While cordierite monoliths do not have a very high surface area (typically less than 1 m²/g), many materials that do have high surface areas can successfully be bound onto it. Cordierite honeycomb monoliths are produced for commercial use, and are in greatest demand for automobile exhaust emissions control. In this application, high-surface area alumina is bound onto the monolithic surface and its integrity is retained throughout tremendous physical and

chemical changes encountered during the countless duty cycles experienced. In contrast, carbosieve S, as a solid monolith, would be its own substrate.

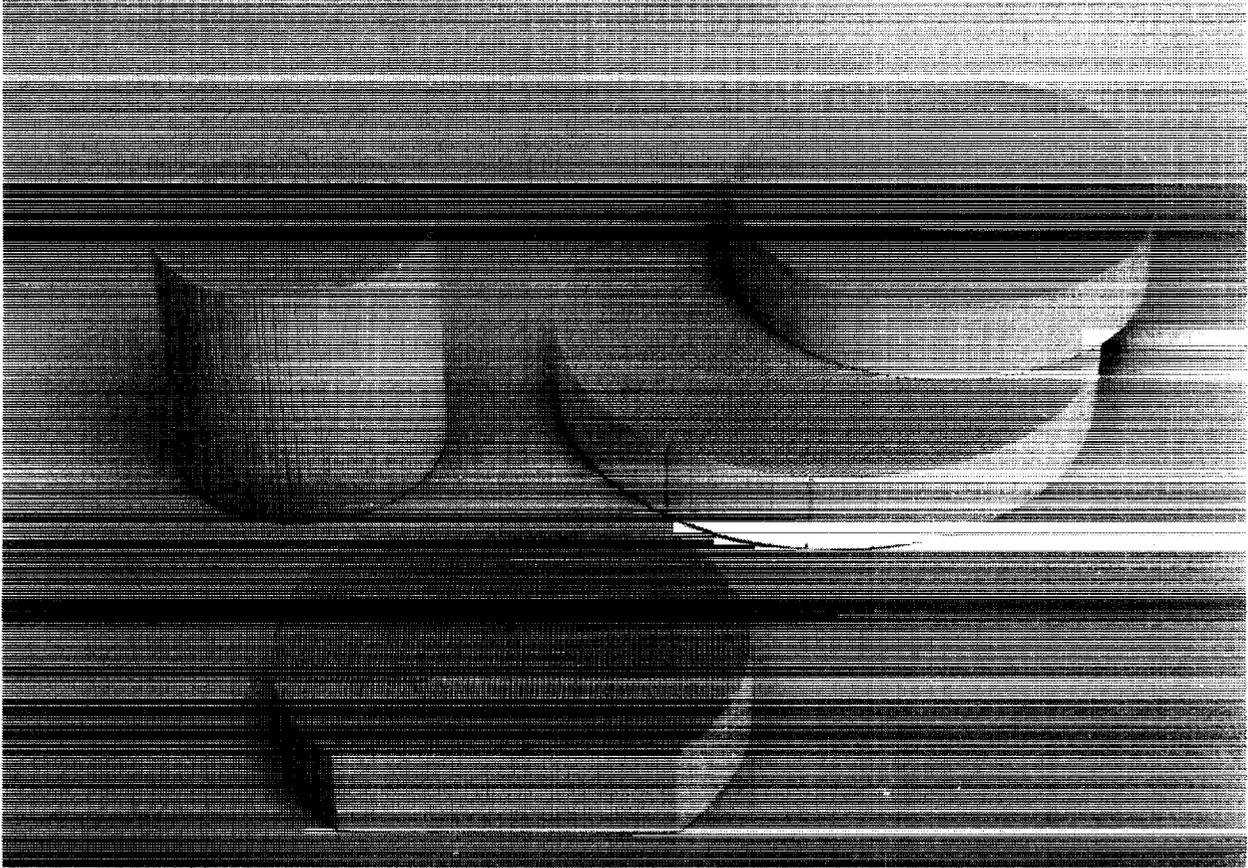


Figure 2-2. Some cordierite honeycomb monoliths.



Section 3

Developmental Testing

The development of the molecular adsorber was executed under a very tight schedule. Accordingly this phase could not include a complete comparative evaluation of all types of adsorbents challenged by every type of contaminant expected inside the instrument. Furthermore time did not permit the production of coatings of each type of adsorbent. Therefore it was decided to select an adsorbent for further evaluation on the basis of a straight forward capacity test. These capacity tests were conducted with various molecular sieve beads, commercially available, and with chemicals acting as surrogates for WFPC-2 contaminants.

The four zeolites and one nonzeolite adsorbent in the capacity tests were selected on the basis of structural differences (e.g., pore size and dimensionality of internal structure), as discussed, and past experience in their use as adsorbents.

The chemicals were chosen to represent species that test the ability of adsorbents to adsorb, including a range of molecular sizes and functional groups, and molecular weights consistent with the species present in the outgas from instrument materials and subsystems.³ The choice of sorbates was also based on similarity to suspected contaminant compounds' volatility, molecular structure, and polarity. Benzophenone is large in cross section and due to its composition possesses a weak, but symmetrical, dipole character. Benzylimidazole is nearly planar but unsymmetrical, large and with a strong but unsymmetrical polarity. Imidazole is small and has the same composition as benzylimidazole but without the bulky aromatic ring attached and with a more nearly symmetrical polar character. (This compound was chosen to evaluate the effects of the aromatic ring attached to the polar ring and determine shape selectivity importance in sorbent choice.) Benzylisocyanate resembles plasticizer contaminants used as catalysts for binding. N-tetradecane was chosen because of its contrasting long chain, alkyl composition and commensurate non-polar character.

As a result of these tests faujasite (e.g., 13X)¹² was selected. This zeolite has a silica-aluminate "cage," three-dimensional open crystalline structure, with large "cages" and smaller connection channels. It also has a large available surface area and a structural geometry that is conducive to molecular shape selective trapping. Finally, it has various sites that are polar in nature, an important feature for the retention of chemical compounds that possess polar functional groups or in which polarization is induced.

Because of the concern about the release during flight of water adsorbed during ground handling, a synthesis of dealuminated zeolite (DAZ) was attempted. DAZ was expected to be a "waterproof" enhancement of zeolite, which

would not adsorb water. The DAZ beads, successfully prepared, were further evaluated, along with 13X.

The sticking coefficient tests were considerably more difficult and thus conducted only on the best candidate from the capacity tests. In these tests, real WFPC-2 sources, representative of the two most serious items, electrical windings and cables with connectors, were employed. The outgas rate of these two sources had been previously standardized at the temperatures used.³ The first tests were conducted with zeolite beads.

On the basis of the capacity and sticking coefficient tests, only 13X zeolite (or equivalent) was selected for the development of an appropriate coating procedure on the cordierite honeycomb substrate. Once again, the number of reasonable processes and process parameters was much too large for an exhaustive evaluation. Additionally, a formal vibration test with a complete particle shedding measurement would require more resources than were available. Therefore, the matrix of candidate processes and parameters was culled on the basis of experience. Small (1 in. x 1 in. x 0.5 in. deep) coated molecular adsorbers were prepared from the foreshortened matrix of candidates. Some of the binding approaches were then eliminated through the examination of the integrity of the coatings by scanning electron microscopy. The relative particle shedding of adsorbers coated by these procedures was measured in a simple direct screening test described below. The capacity of the three best adsorber types was tested to ensure that the coatings retained an adequate capacity compared to beads. Sticking coefficient tests were conducted on these adsorbers to select a baseline and a backup coating. These two specific approaches were carried through proof of concept and formal vibration qualification testing later.

3.1 Adsorbent Bead Testing

3.1.1 Capacity Tests¹³

The capacity tests comprised the exposure of a weighed sample of molecular sieve beads to the surrogate chemical compound at a known mass flux (corresponding to a vapor pressure of 100 torr) for a time sufficient to reach a significant fraction of the saturation adsorption. The mass adsorbed was measured approximately by weight increase of the adsorbent and checked by the weight accounting of the chemical. Thermogravimetric analysis (TGA) provided a more precise determination of adsorbed mass, but only the fraction of it that would desorb at the highest temperature that was run on the TGA. The fractional weight change of the exposed sample, corrected for an unexposed sample's fractional weight change, is thus a conservative value (lower limit) of the capacity.

The test apparatus was a 70 ml glass flask fitted with an external heating mantle and a stopper assembly at its top connected by a valve to a mechanical

vacuum pump. A weighed quantity of chemical was introduced into the flask. Preconditioned (dried at 350°C for two hours) adsorbent beads were then suspended in a cage of either clean copper wire or quartz, that hung from the end of the vacuum tubing inside the flask. After a nominal pumpdown, the heater was adjusted to raise the temperature of the flask to the published value corresponding to a vapor pressure of 100 torr for the chemical. During the exposure, about 45 minutes, the cage was typically cooler than the flask by about 65°C. The flask was then allowed to cool to 40°C prior to a backfill with nitrogen. The two temperatures were monitored by thermocouples.

The results of these tests are shown in Table 3-1. For the surrogate chemical compounds and the molecular sieves, the lower limit capacities and the corresponding maximum TGA temperatures employed (in parentheses) are given. On the basis of the first surrogate, benzophenone, the adsorbents carbosieve, 4A zeolite, and mordenite were eliminated. Mordenite was eliminated by the following judgement. Although it may have adsorbed more than 11 percent if measured (desorbed) at 350°C, its retention capability per its high desorption at 200°C is questionable. The performance of carbosieve, with the largest internal surface area

Table 3-1. Adsorption capacity test results

	13X	LZY52	Mordenite	4A	Carbosieve
BENZOPHENONE fw 182.22 MP 49-51°C BP 305°C	11.0% (350°C)	16.5% (350°C)	11.0% (200°C)	3.6% (350°C)	3.4% (200°C)
IMIDAZOLE fw 68.08 MP 89-91°C BP 256°C	10.3% (200°C)	4.8% (200°C)			
BENZYLIMIDAZOLE fw 158.20 MP 68-70°C BP 310°C	7.6% (200°C)	3.8% (200°C)			
n-TETRADECANE fw 198.39 MP 5.5°C BP 252.5°C	13.1% (200°C)				
BENZYLISOCYANATE fw 133.15 BP 101-104°C AT 33 torr	10.1% (200°C)	6.6% (200°C)			

by far, demonstrates importance of pore size, i.e., the limited penetration by large molecules. Most of the test matrix was carried out on Linde LZY52 and 13X zeolites. The advantage of 13X is clear. Some extra tests with beads equilibrated with nominal relative humidity (RH) showed that the lower limit capacity of 13X is not affected by the presence of adsorbed water.

3.1.2 Sticking Coefficient Tests

The sticking coefficient tests consisted of a series of exposures of a counted number of 13X zeolite beads to a known source followed by a measurement of their desorption. The sticking coefficient is represented by the ratio of the adsorbed contaminant mass, as measured during the desorption, to the incident contaminant mass, as measured during the exposure. In all of the tests, the exposures were conducted in a vacuum in the JPL Molecular Contamination Investigation Facility (MCIF)¹⁴ System No. 1 (Figure 3-1). The incident mass fluence of species collectable at -20, -50, and -100°C was monitored by facility quartz crystal microbalances (QCMs) simultaneously operated at those temperatures. This measurement underestimates the *total* mass fluence incident on the zeolite beads because species that can collect at colder temperatures are not accounted for. This limitation may be acceptable because the WFPC-2 surfaces of interest operate no colder than -100°C. The two sources employed were: a collection of four different cable connectors and cable pieces representative of WFPC-2 use; and a representative disassembled electromechanical device. This "cable" source and "motor" source had both been previously well characterized in the MCIF.

Two different techniques for the measurement of the adsorbed mass in the subsequent desorption were employed, thermogravimetric analysis (as in the capacity tests) and QCM measurements (in MCIF System No. 2). Each of these approaches has advantages and disadvantages. The TGA provides a more complete desorption at 200°C than the 150°C limit of the MCIF. (This limitation of the current MCIF may be eliminated in a future upgrade.) Note, however, that this factor can only cause an *underestimate* of the sticking coefficient. The TGA is also faster since an evacuation cycle of several hours is not required. The QCM possesses much greater sensitivity, a distinct advantage for the small masses to be measured. For this reason the exposure of the zeolite beads for TGA desorption had to be "accelerated" as discussed below, which introduced another source of uncertainty in the results. The most important advantage of the desorption in the MCIF is the direct measurement of the same contaminant species of interest that were monitored during the exposure. Thus, the sticking coefficient for species from the source that collect at -20, -50, and -100°C are directly determined. The TGA desorption-measured mass may include light molecular weight species that cannot collect at -100°C and were not monitored in the exposure phase. This factor is not acceptable because it may lead to an *overestimate* of the sticking coefficient. By a similar argument, the desorption of unexposed control beads may be more critical for the TGA approach.

In the actual exposures the zeolite beads were suspended in a clean, bakedout stainless steel mesh at the QCM collection plane (QCM position) and at a location directly above the aperture of the MCIF sample isolation chamber (aperture position). The use of the aperture position was intended to produce beads with a large enough adsorbed mass for the TGA in a reasonable exposure duration and at a source rate such that the MCIF could be readily cleaned for other runs. The ratio of the desorptions from beads at the two positions for the same source provided the acceleration factor needed to interpret the sticking coefficient for the "aperture position." A simple prediction of this factor yielded values in the range 2.2 to 10.4, depending on the assumptions about the mode of transport.

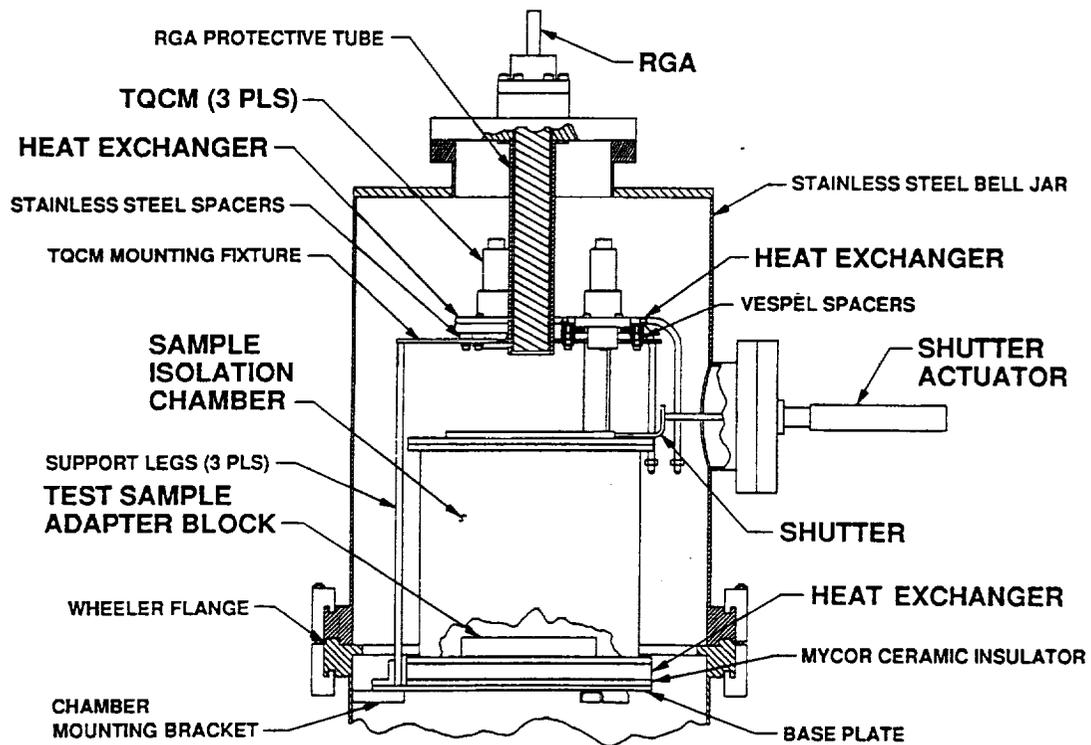


Figure 3-1. The JPL Molecular Contamination Investigation Facility (MCIF).

The earliest tests were concerned with the establishment of a proper procedure. The first test results for an exposure to the cable source at the aperture position in a copper mesh basket yielded 2.5 to 2.8 μg per bead by desorption of part of the beads in the MCIF No. 2 at 100°C (only). For the acceleration factor later determined to be 10.4, the incident exposure fluence had been 110 $\mu\text{g}/\text{cm}^2$ (3.5 μg per bead). Thus, the sticking coefficient for these preconditioned (dry) beads was at least 0.7. The rest of the exposed beads were measured by TGA, which produced the interesting desorption result of 102 μg per bead at 100°C. This discrepancy is due to: the desorption of a significant amount of adsorbed contaminants too light to be collected by the QCMs (or by WFPC-2) but detectable by mass change in the TGA; a non-representative control bead sample for the TGA; and the sensitivity limit of the TGA. A proper control would have corrected for the light contaminants in the unexposed beads, but no control for the amount adsorbed during the exposure is feasible. Since a typical amount of water could be as much as 600 μg per bead (10 percent by mass), this is a serious shortcoming. Finally, the quantity of actual interest (ca. 3 μg per bead) corresponds to a fractional mass change of only 0.05 percent, which is at best at the TGA limit, notwithstanding the large corrections required.

A second test, conducted with the motor source and the beads in a quartz basket, produced similar results. The sticking coefficient by QCM desorption was at least 0.74 (112 ng per bead desorbed at 100°C out of 152 ng incident). Here the acceleration factor was later determined to be only 2.2. Because this factor and the signal sought were so small, the TGA control correction was statistically invalid for the measurement of about 100ng. The best estimate by TGA of the mass accumulated in the exposure (only) and then desorbed is 130 μg per bead at 100°C and 7.1 μg additional at 200°C, subsequently. Most of this mass apparently was water.

In the final tests with beads, the beads were exposed at both locations and desorbed only in MCIF No. 2. The TGA was eliminated. Also these beads were equilibrated with room ambient conditions to provide a more realistic challenge to contaminant adsorption; that is, to address the possible interference due to previous adsorption of large amounts of water. Finally, the desorption cycle was established as: 7 hours at +100°C, a 1.5 hour transition to +150°C, and 4 hours at +150°C. The time required for this measurement, in addition to a pumpdown, is long. Even so, the raw data indicated incomplete desorption. The desorption, as collected at -100°C, of 13X beads exposed to the motor source in the aperture position showed nearly complete desorption in 7 hours of species that desorb at +100°C and incomplete desorption of other species at +150°C after 4 hours more (Figure 3-2). Therefore all sticking coefficient values are lower limits.

The results of these sticking coefficient tests are shown in Table 3-2. In all cases the values are at least 0.5 for all contamination collectable at -100°C, as determined by the most complete desorption, which is satisfactory for the intended application. The values for each source at the two positions are consistent, as they should be. The data also exhibit some other interesting features that suggest further

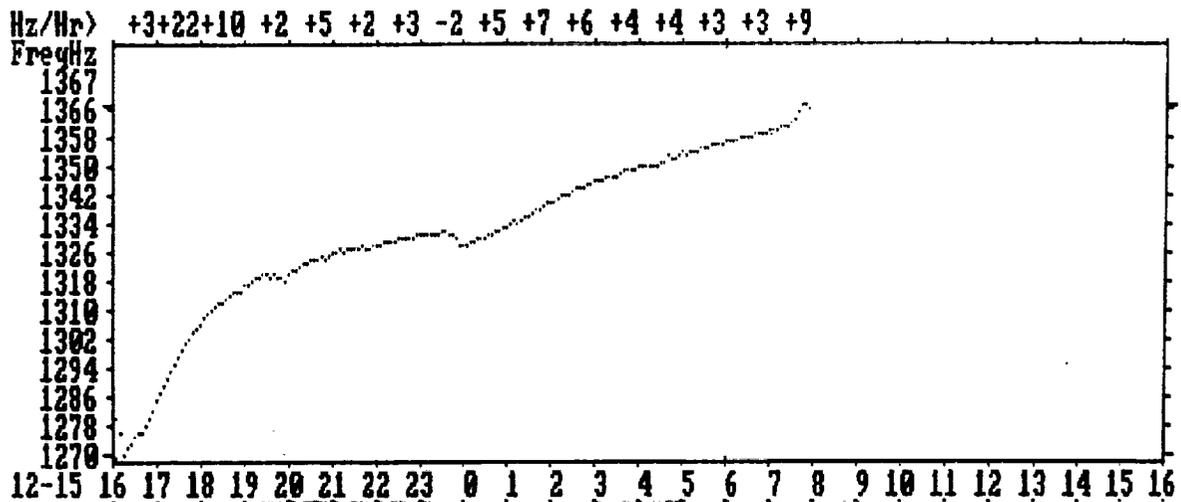


Figure 3-2. The desorption of 13X zeolite beads. Shown is the collection rate (Hz/hr) by a CQCM at -100°C for the beads at 100°C for 7 hours, during a transition to 150°C for 1.5 hours, and at 150°C for 4 hours. The contaminants were originally adsorbed from the WFPC-2 motor source.

research would be useful. For example, the data show that most of the adsorbed contamination from the cable source is desorbed at +100°C in four hours versus the entire cycle, while a much smaller fraction for the motor source desorbs at +100°C. This is consistent with a stronger adsorption (energy) of the motor source species, which are also less volatile (as shown by their efficient collection even at -20°C collection temperature). However, there is evidence that the sticking coefficient is smaller for the less volatile fraction (per collection temperature). The noise in this experiment at poor statistics and the difficulty of experimental controls are revealed when more mass is desorbed than incident.

The DAZ was dropped from further consideration at this time because the bead material as synthesized contained an unacceptable amount of contaminants collectable at -100°C, as measured by unexposed controls in the sticking coefficient tests. This decision was made under time pressure. The process had *not* been optimized, but merely successfully demonstrated.

Table 3-2. Zeolite beads sticking coefficient results

Source	Position	Collect Temp °C	Expose Mass* ng	Desorb Cycle	Desorb Mass ng	Net** Desorb Mass ng	Stick Coeff
Cable	QCM	-100	107	+100°C	75	42	.39
		-100	107	all***	119	60	.56
		-50	1.65	all	20	12	n/a
		-20	n/a	all	12	7.0	n/a
	aperture	-100	1100	+100°C	374	341	.31
		-100	1100	all	655	596	.54
		-50	17	all	140	132	n/a
		-20	n/a	all	100	95	n/a
Motor	QCM	-100	23.8	+100°C	47	14	.60
		-100	23.8	all	100	41	n/a****
		-50	16.5	all	14	6.0	.36
		-20	10.6	all	9.4	4.4	.42
	aper	-100	52	+100°C	70	37	.71
		-100	52	all	97	38	.73
		-50	36	all	16	8.0	.22
		-20	23	all	9.4	4.4	.19

* All masses expressed per zeolite bead. Bead mass approximately 6 mg.

** Net of desorption from unexposed beads. These unconditioned beads desorbed 33, 59, 8, and 5 ng per bead at +100°C for four hours collected at -100°C and for the entire desorption cycle collected at -100, -50, and -20°C, respectively.

*** The entire desorption cycle was 100°C for 7 hours, a 1.5-hour transition to 150°C and 4 hours at 150°C.

**** This result is taken to mean a probable sticking coefficient of 1.0.

3.1.3 Other Tests

A mass spectroscopy study of the desorption from beads as a function of temperature was conducted to evaluate the release of water and the retention of adsorbed contamination from 13X zeolite.¹⁵ The spectra show that some water was released from unexposed (as received) 13X beads at 50°C, as well as carbon dioxide

and some other species represented at 40 to 45 amu. Beads saturated with benzophenone did not desorb it readily until 200°C. Additional beads exposed in the sticking coefficient tests were desorbed into the mass spectrometer also. The cable source contaminants apparently did not easily desorb until almost 200°C and appeared at 40 to 45 amu. On the other hand, the contaminants from the motor source began to desorb at 75°C, with features near 40 and 60 amu. This large apparent difference in desorption characteristics may be due to differences in the actual species adsorbed from these two sources or may possibly be due to a decomposition, during the desorption, of the adsorbed species from the motor source. These results may also explain the smaller, apparent sticking coefficients cited above for the cable source (i.e., incomplete desorption).

Similar tests in the MCIF No. 2 with beads exposed to the cable source showed that for these fairly dry beads, no significant increase in water desorption occurred until 100°C, per the residual gas analyzer. The apparent water desorption rates from 40 zeolite beads (ca. 160 mg) were approximately 50 and 90 mg/hr at 100 and 150°C, respectively. At both temperatures these rates dropped below detectability over background in a few hours.

For contaminants collectable at -100°C, the desorption as a function of temperature and time is given in Table 3-3, expressed as a fraction of the total desorption observed and of the incident mass fluence. For the entire desorption cycle, the latter corresponds to a sticking coefficient of 0.7, better than the previous result above for the cable source, but for a duration at 150°C twice as long. The retention of adsorbed contamination was acceptable; very little desorption occurred at temperatures at or below 45°C, the flight acceptance (FA) hot temperature for WFPC-2.

Table 3-3. Fractional desorption of cable source contaminants from 13X

Temperature, °C	Duration, hr	Fraction of Desorbed	Accumulative Fraction of Desorbed	Fraction of Incident	Accumulative Fraction of Incident
25	3.75	0	0	0	0
35	4	.09	.09	.06	.06
45	4	.04	.13	.02	.08
100	6.5	.12	.25	.09	.17
150	7	.75	1.00	.52	.69

3.2 Adsorbent-Coated Substrate (Molecular Adsorber) Testing

Once 13X or equivalent zeolites had been selected as the adsorbent, an acceptable procedure for coating the cordierite honeycomb had to be developed. This activity proceeded through both analysis and trial and error. The minimum desired coating thickness was calculated based on estimates of the total amount of contaminant molecules that could conceivably be released for capture and the amount of sorbent that could be bound upon the honeycomb monolithic supports. Various approaches to binding and different forms of faujasite were employed to fabricate real samples. Visual inspection and some microphotography provided the list of candidate processes for further testing.

3.2.1 Particle Shedding Screening Tests¹⁶

The particle shedding screening tests were conducted on shock test apparatus comprised of a pivoted test tube clamp, a vertical stand from which it could pivot, weights to cause the clamp to fall toward the stand base (table), and rubber stoppers to brake the fall and induce the shock. The clamps held molecular adsorber test samples between foam cushioning. The shedding was directed toward an upward facing collection surface on the table, when the clamp and the sample were braked before the sample could hit the collector. Both Petri dishes and the fallout plates for a Uramec Particle Fallout Photometer (PFO) (model 255) were employed. The apparatus was "calibrated" with an accelerometer to reproducibly provide about 80 G peak acceleration (compared to the qualification peak level of 100 G).

Nominal 1 in. x 1 in. x 0.5 in. pieces of adsorber (zeolite-coated cordierite or z-cord for short) were evaluated for their *relative* shedding in these screening tests. A list of the types of coatings is provided under the heading "designation," in Table 3-4. The protocol consisted of three shocks with one open honeycomb face pointing down at the collector, followed by one shock with the opposite face down. The last inversion was intended to collect particles that may have migrated up the honeycomb cells during the three prior shots.

The photometer provided simple, fast, and inexpensive relative readings of the particle shedding. The fallout plate needs only to be inserted into the instrument to obtain a reading. The measurement, in PFO units, is related to the total integrated scatter of white light at grazing incidence to the black glass plate surface. The area sampled is about a 15-mm-diameter circle. A reading of each serialized plate when clean yielded a very small background correction (typically 4 PFO units). However, an optical image analysis provided direct values of the fractional area covered by particles (obscuration ratio), particle size distributions, and a permanent computer record. A larger or smaller area may be analyzed; 12 fields of 2 mm x 2 mm were employed. There are disadvantages. The collection plates have to be taken

to this system. The cost of (and the wait for) a useful optical image analysis is significant, about a man-day.

The screening began with the shock testing of one sample of each candidate adsorber into a Petri dish and a visual inspection. On this step, both washcoats involving calcium hydroxide were eliminated on the basis of extensive shedding during the handling to clamp the samples into the apparatus. The 13X on alumina 2:1 and on alumina 3:1 (AL)¹⁷ were eliminated by a visual inspection of the fallout. Then the four remaining of the first eight candidates were image analyzed. The 13X/alumina 3:1 was eliminated on the basis of two large particles on the image analysis of the Petri dish. (Note that it is still much cleaner than the earlier rejected candidates.) The 13X/CaCO₃ was kept because other than one particle, the PFO plate looked good. The 13X/Ludox 30, which had been average on the first test into a Petri dish, virtually shedded nothing on a second test into a PFO plate. It was retained as a possible in the hope that the good version could be reproduced. Finally, the image analysis of the Petri dish for Y/alumina 2:1 (a specific synthetic 13X type faujasite on an alumina washcoat)¹⁸ yielded the best (lowest) value. Note that a correlation of three points between obscuration ratio and PFO units has been provided in Table 3-4. This is a small contribution to a currently sparse data set.

At this point there were only three candidates for further study, including one that was in some ways as bad as the ones rejected and one that was the best by far but exhibited extreme variability. Therefore, a zeolite preparation¹⁹ that was deposited directly on acid-leached cordierite, believed to be a good candidate (by optical microscopy), was added to the list. New 1 in. x 1 in. specimens of each were prepared, enough identical pieces to supply a variability screening test of four replicates through the shock test protocol and also for the capacity and sticking coefficient measurements. Somewhat thinner coatings were attempted. The results of the variability screening tests are given in the last column of Table 3-4. A range of values corresponding to repeated readings with the PFO is shown for each of the four replicates tested. On this basis the CaCO₃ washcoat was rejected. It should be noted that some of the combinations of materials and procedures that were not selected may be worth further investigation.

Table 3-4. Shedding evaluation of molecular adsorbers

Designation*	Comment	Initial Screening			Variability Screening
		PFO	Obscuration Ratio		PFO
		Units	PFO Plate	Petri Dish	Units
13X/Alumina 3:1	eliminated; 2 large particles on petri	527	2.1×10^{-3}	2.9×10^{-2}	
13X/CaCO ₃		1692	7.7×10^{-3}	1.2×10^{-2}	off scale 1915-o/s off scale 1185-1400
13X/Alumina 2:1	eliminated; high shed initial screen				
13X/Ludox 30	initial screen variable	79	7.4×10^{-4}	1.4×10^{-2}	490-500 385-390 390-403 547-620
13X/Alumina 3:1(AL)	eliminated; high shed initial screen				
13X/Ca(OH) ₂ 3:1	eliminated; shed on handling to screen				
Y/Alumina 2:1		n/a	n/a	6.4×10^{-3}	293-402 500-520 275-286 34-65
13X/Ca(OH) ₂ 3:1(AL)	eliminated; shed on handling to screen				
Zeolite Prep (AL)	added to list to replace 13X/Alumina 3:1 when eliminated	n/a	n/a	n/a	166-184 130-160 405-425 300-336

* Adsorbent/washcoat (if any). (AL) denotes cordierite was acid leached before further processing.

3.2.2 Capacity Tests

Capacity tests on the final three candidate molecular adsorbers (13X/Ludox 30, Y/Alumina 2:1, and zeolite prep (AL)) were conducted with tetradecane analogously to the tests described above on 13X beads. As expected, the capacity as a fraction of zeolite mass was little changed. The measurements showed a capacity of 1 percent by weight of adsorber (coated cordierite). Later measurements indicated that the thinly coated adsorber is 8 to 10 percent zeolite by weight, so that on a zeolite mass basis, the measured capacity was 10 to 12.5 percent (versus 13.1 percent in Table 3-1 for beads). This change is of no engineering significance.

3.2.3 Sticking Coefficient Tests

The same three final adsorber candidates were exposed simultaneously to the "cable" source in a test analogous to the previous tests on 13X beads. The z-cord specimens had been preconditioned, as was intended for flight units, by an overnight bake out in air at 250°C and then a nitrogen purge. Two pieces approximately 0.5 in. thick of each type were stacked in square cross section (1 in. x 1 in.) quartz tubes with tabs to retain the adsorber. The three quartz tubes were suspended in the MCIF No. 1 at the QCM position. A comparison of the total desorption from the bottom (front) piece and from the top (back) piece was planned to provide direct evidence on the matter of the utility of 0.5 in. versus 1 in.-thick molecular adsorbers. Unfortunately, a small amount of contamination may have entered the top (back) piece. The exposure, directly onto the bottom pieces' faces, was 3800 ng/cm² collected at -100°C in about 24 hours. ("Cable" source was at 40°C; chamber pressure was 3 to 6 x 10⁶ torr.)

The exposed specimens were then desorbed (3 hours at 45°C, 6 hours at 100°C and 10 hours at 150°C) one at a time in MCIF No. 2. The results in Table 3-5 show sticking coefficients for species collectable at -100°C in the range 0.53 to 0.75. The observed incomplete desorption at the maximum available temperature means that all the values are lower limits. At the end of 10 hours at 150°C, the desorption of the Y/Alumina adsorber, for example, was continuing linearly at a rate that was adding 0.02 per hour to the measured value of the sticking coefficient. Because of the adsorber geometry, the values are expected to be larger than the values for the "cable" source with zeolite beads (Table 3-2). This expectation was found only for the Y/Alumina adsorber.

Contaminants from the "motor" source, which were collected more readily by the beads, would be expected to be collected extremely effectively by the adsorber. (This test was not conducted due to schedule constraints.) This contention is also supported by the analysis of the contamination from the "cable" source collectable at -50°C. Here the sticking coefficient inferred from the data is 1.0 (not shown). Although these species (collectable at -50°C) are thought to have relatively high molecular weights (like the "motor" source) and to be easily adsorbed, they must

also desorb (or decompose and desorb) readily at 150°C, because virtually all was desorbed.

Table 3-5. Sticking coefficient of molecular adsorbers

Molecular Adsorber	Front 0.5 in.	Back 0.5 in.	Total
Y/Alumina 2:1	0.33*	0.42	0.75
Zeolite Prep (AL)	0.15	0.39	0.54
13X/Ludox 30	0.10	0.43*	0.53

* This result was corrected for a known test system partial failure.

Some of the results were corrupted due to the vagaries of liquid nitrogen delivery (which interrupted the supply to the test). Such results have been approximately corrected and are so noted in Table 3-5.

Despite the difficulty of the attempted test and the related uncertainty in the precise values, the tendency of the back piece to collect about 40 percent of the contamination incident on the front piece is real and at odds with expectation. A colleague, Thomson²⁰ has employed a contamination analysis program (CAP) to predict the adsorption of a contaminant with a flat plate sticking coefficient of 0.5 as a function of depth into the molecular adsorber (i.e., in the sense of the 1 in. honeycomb thickness). He concluded that most of the contaminant is adsorbed in the first 30 mils and that no more than 0.1 in. is useful. While the authors are sure that his analysis was conducted correctly, some possibility exists that the transport of molecules within the adsorbent dramatically changes the situation in a way that the model cannot treat. The environment in the MCIF may be inadequately understood, as well. The resolution of this dilemma had to be deferred to a later time.

For conservatism and in the absence of tight weight constraints, the 1 in. thickness was taken for the baseline. (The minimum thickness to use without further mechanical analysis was 0.5 in.) The Y/alumina was selected as the baseline process, with the zeolite prep (AL), the same adsorbent, as backup.

3.2.4 Retention of Adsorbed Contaminants

The low temperature phase (3 hours at 45°C) of the desorption cycle in the sticking coefficient tests of the molecular adsorbers was used to confirm the adequate retention of adsorbed contaminants. The observed desorption was typically complete. However, the retention is difficult to quantify because: chamber background interfered with the small desorption rate; and some desorption from an *unexposed* (to the cable source) adsorber may also be desorbed at 45°C and collected

at -100°C. By an analysis analogous to the method that yielded the results in Table 3-3 (for 13X beads), the maximum desorption at 45°C as a fraction of the total observed desorption ranged from 0.06 for 13X/Ludox to 0.10 for Y/Alumina. These values agree well with the value for 13X beads, 0.09 (Table 3-3).

3.2.5 Water Desorption

The water desorption at room temperature of a flight-like sample of Y/Alumina adsorber was measured in a dry gaseous nitrogen flow (0.5 liter/minute) with a chilled mirror dew point system (General Eastern model 1311 DR optical dewpoint sensor and Hygro M1 optical dewpoint monitor). The dew point results for a 0.5 in. diameter x 1 in.-long specimen equilibrated at 30 percent relative humidity are shown in Figure 3-3. An artifact of this measurement is that the measured dew point can never be lower than that of the supply nitrogen, -70°C. As will be seen, this is sufficient for the present purpose. The dew point is directly (but not simply) related to the partial pressure of water vapor in the flow, P. The source flux Φ is then given by:

$$\Phi = \frac{\mu P}{A R T} \left(\frac{dV}{dt} \right) \quad (1)$$

where μ is the molecular weight of water, R is the ideal gas constant, T is the absolute temperature of the water vapor (or the nitrogen), dV/dt is the volume flow rate, and A is the presented area of adsorber (corresponding to the 0.5 in. diameter).

The derived source flux versus time at room temperature is shown in Figure 3-4, with the caption "30% RH GN2." (The other data are discussed under PROOF-OF-CONCEPT AND QUALIFICATION TESTING, below.) This technique, although not performed in a vacuum, has the advantage of permitting an upper limit estimate of the total water released during the test. In 96 hours, 0.96 g of water was removed from 1.93 g of adsorber (0.23 g of adsorbent). This result would indicate an initial water loading of 30 percent by weight of adsorbent.

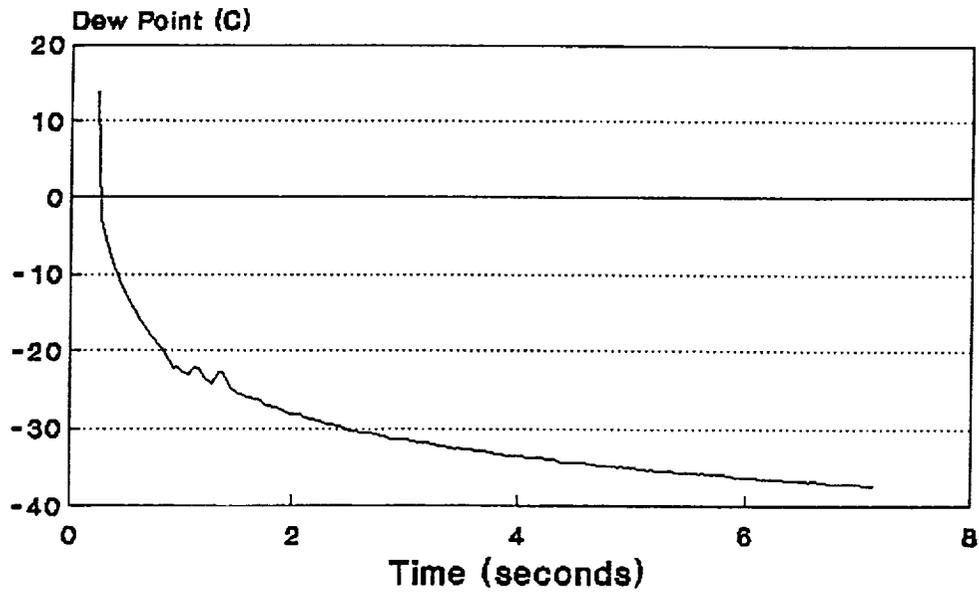


Figure 3-3. Molecular adsorber water emission shown as dew point versus time for a specimen equilibrated at 30 percent RH as measured with a dew point monitor.

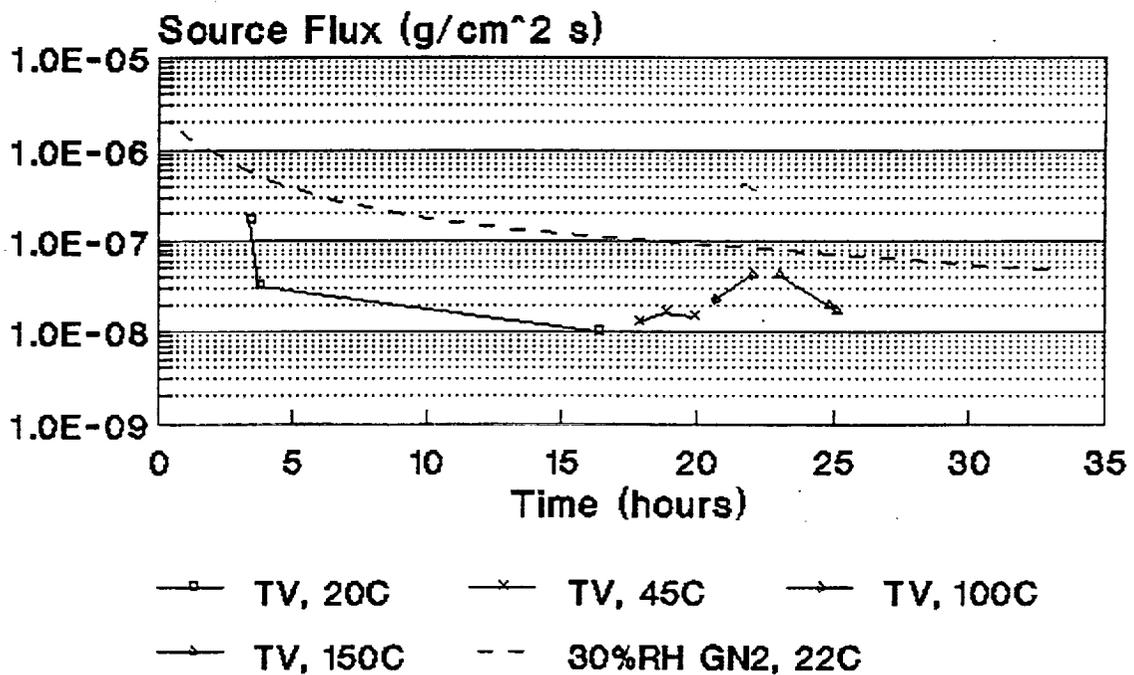


Figure 3-4. Molecular adsorber water emission shown as source flux versus time: for a specimen equilibrated at 30 percent RH, as measured with a dew point monitor (derived from data of Figure 5); and for a specimen exposed to the system thermal vacuum test, as derived from residual gas analyzer (RGA) data for indicated temperatures.

Section 4

Final Design

The final design involved the selection of the flight adsorber, which includes the procedure for its fabrication, and the design of the flight hardware in which the adsorber is mounted, which includes the assembly procedure. The adsorber selected was Y/Alumina 2:1, a Y synthetic faujasite¹⁸ deposited on an alumina washcoat on a cordierite honeycomb. The rationale for this choice has been presented above. The detailed manufacturing procedure is documented as part of the quality assurance records for the WFPC-2 molecular adsorber. The manufacture involved batches of four adsorbers each in a mostly manual process. Some special handling fixtures were also designed and fabricated.

The zeolite coatings obtained by the fabrication procedure were approximately 20 μm -thick, with excellent local uniformity and polycrystalline character, as shown in Figure 4-1. Some isolated imperfections were observed by microscopy, Figure 4-2.

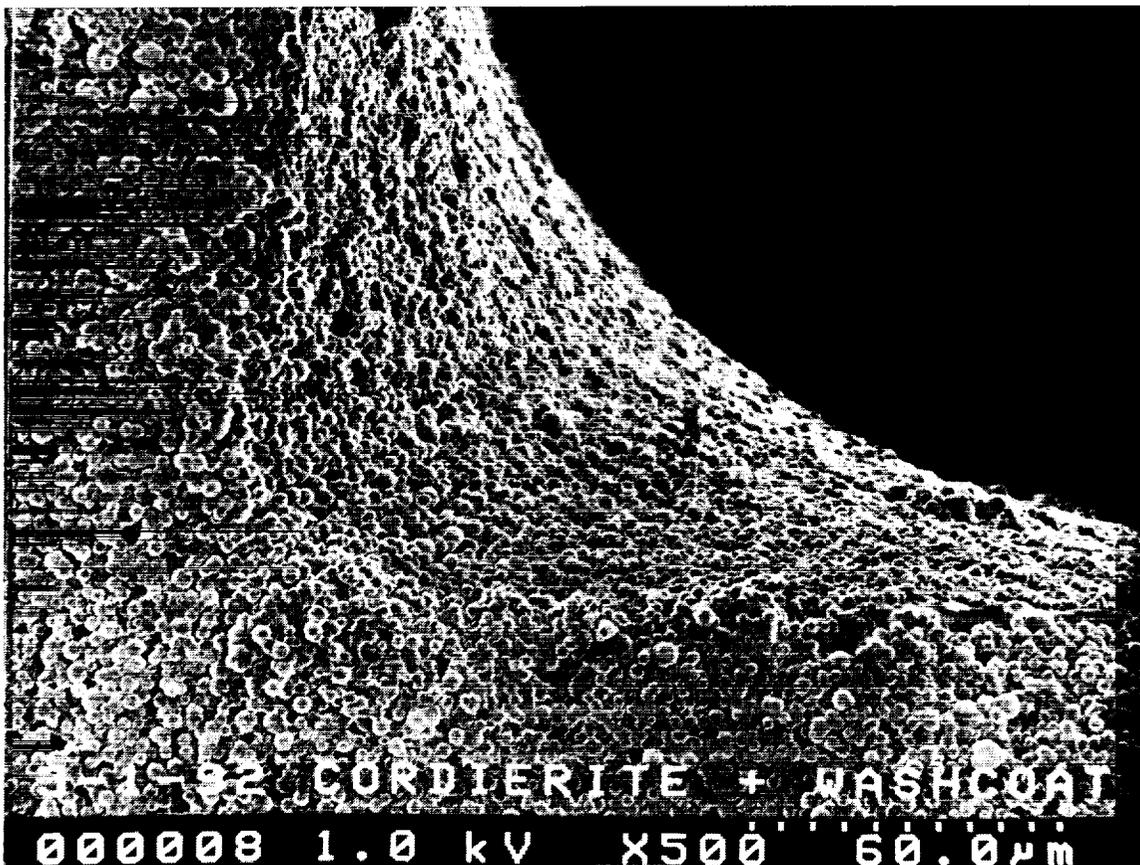


Figure 4-1. Microphotograph (500X) of molecular adsorber coating at corner of cordierite honeycomb cell, showing zeolite crystallites.



Figure 4-2. Microphotograph (200X) of molecular adsorber coating at corner of cordierite honeycomb cell.

Two types of mounting hardware were designed²¹: for molecular adsorber assemblies to be located on the inside of the top and bottom cover plates of the instrument, designated cover units; and for molecular adsorber assemblies to be located on the camera bulkhead in locations vacated by the reduction of the number of cameras from 8 to 4 for WFPC-2 (compared to WFPC-1), designated bulkhead units. Assembled flight units are shown in Figures 4-3 and 4-4, respectively. The frames are aluminum (6061-T6). The cover unit has a 2.5 in. x 4.5 in. adsorber, an adsorber presented area of 52 cm² (1.8 in. x 4.5 in.) and a footprint 4.14 in. x 4.9 in. Its height is 1.37 in., and it weighs approximately 345 g. The rectangular design is appropriate to utilize the available space on the covers with a large presented area for each cover unit. However, this design required a specially ordered (not commercially available) monolith with an extra thick external wall for support.²² The corresponding values for the bulkhead unit are: 3 in. diameter adsorber (stock monolith), presented area 26.6 cm² (2.25 in. diameter), irregular footprint (fits within 4 in. x 4 in.), height 1.43 in., and weight 260 g. Although the two types obviously differ in the shape of the adsorber and the mounting details, another difference should be noted. In addition, the cover unit is open on the face opposite its mounting

surface. The bulkhead unit looks through a hole in its mounting surface (and through the camera bulkhead into the optical bench).

The principal challenge of the design was to clamp the adsorber tightly enough to avoid the generation and release of particles during launch vibration (e.g., by abrasion), without overstressing the cordierite honeycomb. For this purpose, special bow-shaped ribbon springs were designed²³ to fit (with free ends) into rectangular detent slots on the mounting hardware. The locations are the underside of the two flanges of the cover unit that overhang the honeycomb (three on each flange) and the bottom of the "cup" of the cover unit (three on each unit in a triangular pattern). These springs were fabricated from 0.375 in. x 4.5 in. x 0.022 in.-thick CRES 15-5 PH. The design also provided for shims between the honeycomb and arch of the springs to correct for the thickness variation of the flight honeycomb to achieve the proper clamping force. The required tolerance (ca 10 mils) was small compared to the uniformity of the cut cordierite. Therefore, each flight unit was shimmed per a calculation with its premeasured thickness. The selection of the side of the honeycomb to locate the springs was driven by a desire to keep one of the two pieces that comprise one unit's frame (namely the mounting surface or base plate) a simple stock flat plate. This choice also simplified assembly because the springs and shims could be "dropped in" first, then the adsorber, and finally the base plate fastened on top, last.

Other features on the cover unit include captive nuts for fastening by machine screws through a thin wall without risk of dropping a nut inside WFPC-2. (The bulkhead units are fastened by screws into inserts on the bulkhead.) The pan heads of the cover unit assembly screws also intentionally protrude into the mounting plane to suppress corner slapping during vibration of the thin WFPC-2 cover.

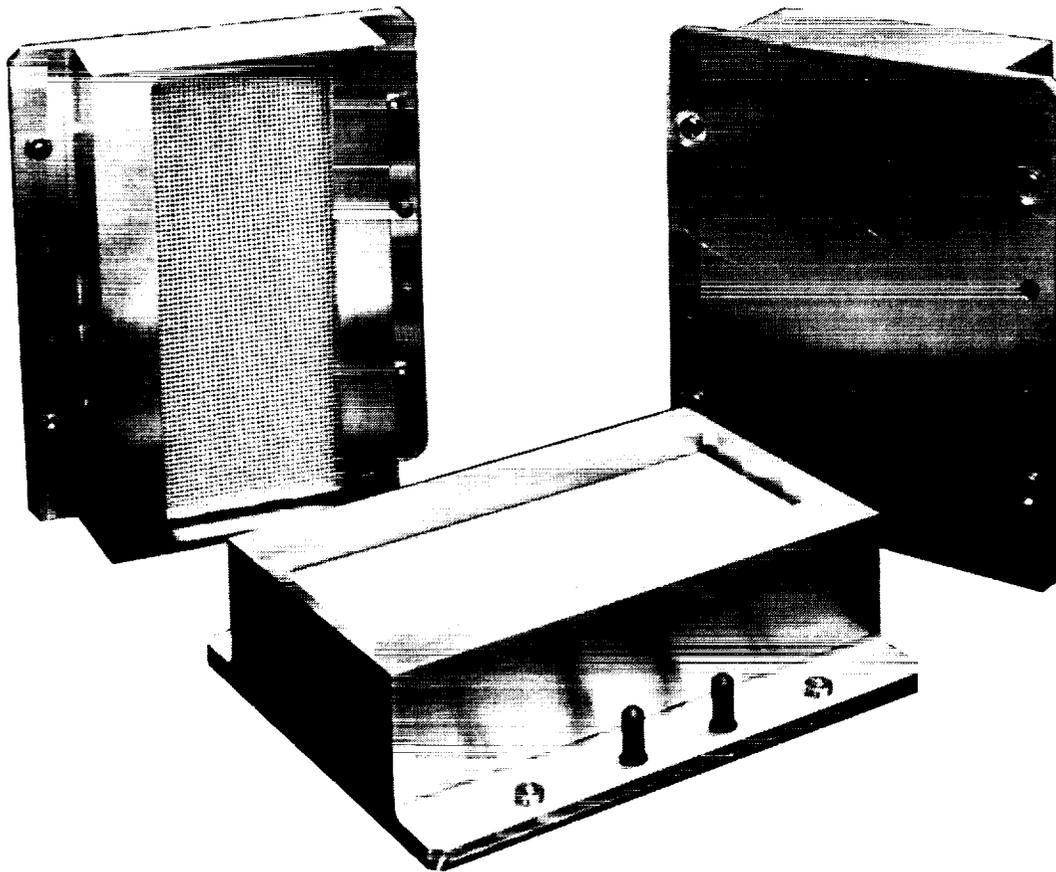


Figure 4-3. Flight cover molecular adsorber assemblies.

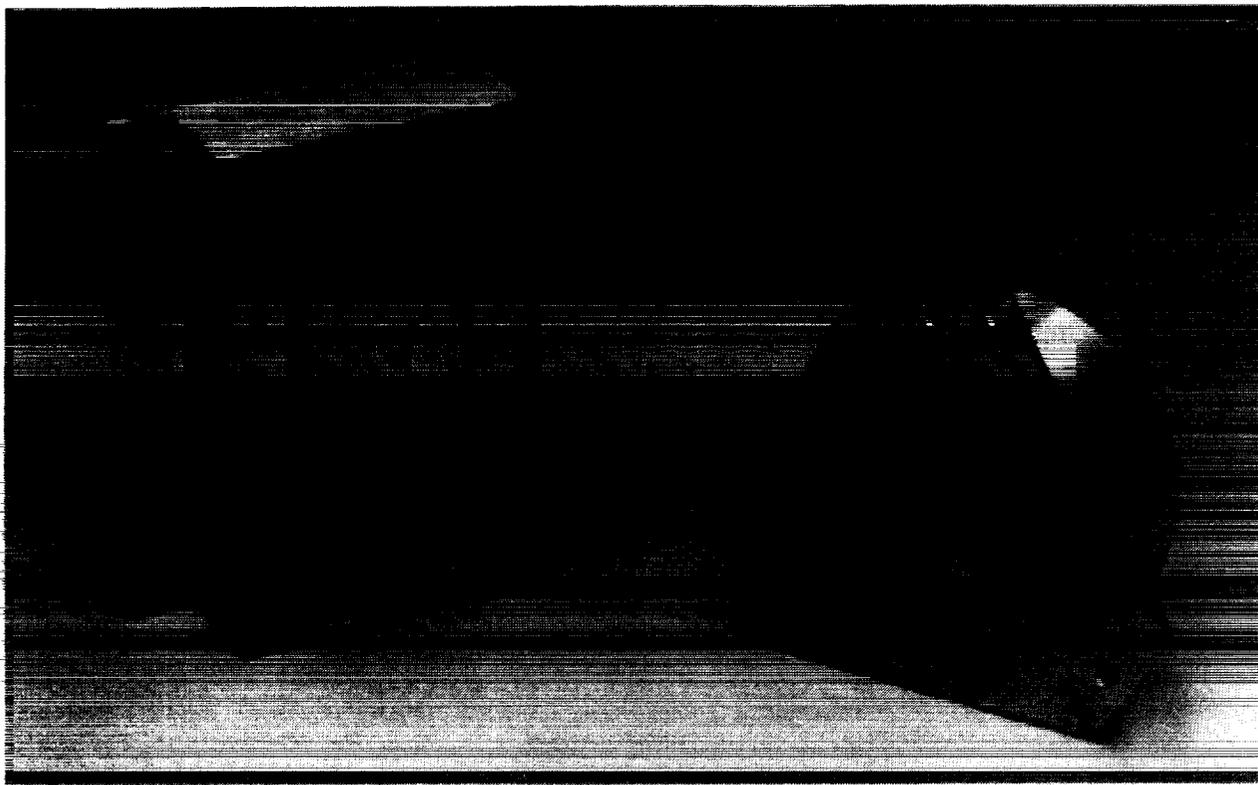


Figure 4-4. Flight bulkhead molecular adsorber assemblies.

Section 5

Proof-of-Concept and Qualification Testing

5.1 Particle Shedding Qualification

As discussed, the major challenge to the use of a zeolite-based molecular adsorber for a flight instrument is the potential for particle shedding during the launch vibrational environment. Thermal cycling in a vacuum was also explicitly considered. The qualification of the molecular adsorber comprised an analysis for the permissible quantity of particles, the design and fabrication of a design test model (DTM), and the actual test program.

The permissible amount of shedding was established through a worst-case analysis of the transport of the particles to the WFPC-2 optical surfaces²⁴ and a comparison with the existing requirement for those surfaces. The shedding requirement was based on the arbitrary but reasonable proposal that the molecular adsorbers increase the obscuration ratio²⁵ of the optics by no more than 2.8×10^{-5} . This value is less than the expected obscuration ratio at pre-ship, 4.2×10^{-5} , and at launch, 1.4×10^{-4} . The launch, deployment and installation of WFPC-2 are expected to add only 3.3×10^{-5} (mostly from the adsorbers).²

As a convenience, the quantity of particles per unit presented surface area of an adsorber may also be expressed as an obscuration ratio. The value of the obscuration ratio is equivalent to that of a "contact print," i.e., a collecting surface of the same area as the adsorber. The transport factors determined from the analysis were essentially unitless dilution factors. The results were: 3.7×10^{-3} for the cover units and 2.2×10^{-3} for the bulkhead units.²⁴ The more stringent (smaller) value was approved for the DTM test.²⁶

The DTM design was intended to be as close as possible to the design of the flight cover unit. However, due to a misunderstanding about the mounting clearance available on the instrument's cover, the DTM unit was designed and fabricated with a 3.5 in. x 4.5 in. adsorber. Thus, the DTM was 1 in. wider in the direction perpendicular to the mounting flanges' long dimension than the flight cover units. (See Figure 4-3 for a flight cover unit.) By similarity, the shedding from this larger assembly, with a larger span between the clamping surfaces and a larger clamped mass, could only be greater than from the flight cover unit. Therefore, the otherwise identical DTM was sufficient for purposes of qualifying the design. By a similar analysis, the flight cover unit was a worse case than the bulkhead unit.

In order to measure the particle shedding during either a vibration test or a vacuum thermal cycle test, the DTM base plate had a 0.75 in. hole on center to act as a view port to allow any particles that were released or generated to fall onto an upward facing piece of 1 in. wide pressure sensitive tape. The tape used and the

procedure for counting the particles collected were compatible with a published procedure for the direct measurement of surface particles by lifting them from the surface with a piece of tape.²⁷ Specifically, the particles' obscuration ratio was directly determined with an image analyzer in terms of pixel counts.²⁸ Additionally, the particles found on the base plate after the completion of all of these tests were rinsed off and counted separately.

For the vibration tests, the tape was placed over a matching hole in the vibration test fixture plate on its bottom surface after the DTM had been mounted to the top of the plate. The bottom of the fixture plate also had a milled channel so that the tape would not be compressed when the fixture plate was fastened tightly to the shaker table. The location of the tape permitted the tape to be changed between each axis of vibration, while the fixture with the DTM mounted to it was unbolted and moved. Thus, the shedding from each axis was separately measured, except for the particles on the base plate. Another tape was placed on the bottom of the base plate prior to the mounting of the DTM on its heat exchanger for the thermal cycle test. During these handling operations and the thermal cycle test, the DTM was protected by a cover against environmental fallout particles.

The recommended test environments, the actual test environments, and the results of the shedding measurements are documented in one report with attachments.²⁹ As a test of the basic design, the DTM with an uncoated honeycomb was vibration tested early in the development. At the qualification level, 15.9 Grms for 180 seconds on each axis, the obscuration ratio was determined to be 7.3×10^{-4} , with negligible particles on the base plate. This was a positive result, especially the lack of significant generation of particles by abrasion, which would have appeared on the base plate. Later, the DTM cleaned per flight procedures and with an adsorber manufactured according to the flight procedures was vibration tested as above and then thermal cycle tested in a vacuum (seven cycles of 1 hour at -55°C and 3 hours at $+45^{\circ}\text{C}$, followed by 1 hour at -55°C and 12 hours at $+55^{\circ}\text{C}$, with $5^{\circ}\text{C}/\text{minute}$ ramps). The results of these tests were a total calculated obscuration ratio of 7.5×10^{-5} (2.5×10^{-4} worst case) for the tapes, to which a contribution from the base plate rinse of 1.4×10^{-4} should be added. The DTM easily bettered its requirement of 2.2×10^{-3} maximum. The data also indicated that the two axes in the mounting plane were far worse than either the other Cartesian axis or the thermal cycle: X-axis, 3.8×10^{-5} ; Y-axis, 3.1×10^{-5} ; Z-axis, 4.0×10^{-6} ; and thermal cycle, 2.2×10^{-6} , respectively. (The axis parallel to the overhanging and the mounting flanges' long dimension is the X-axis.)

As a result of the qualification test, an adequate flight acceptance (FA) test was determined to be a one-axis vibration test at the FA level. Thermal cycling had been shown to be benign. The worst-case axis was known to be in the mounting plane. Specifically for the cover unit, the X-axis was expected to be slightly worse than the Y-axis. To conservatively eliminate the effect of the other two axes, the shedding requirement was reduced by a factor of three from the results of the analysis.²⁶ Again, a view port was needed to collect the downward falling particles on adhesive tape. Therefore, each flight adsorber³⁰ was FA tested in a frame

assembly exactly flight-like except with a view port in the base plate. (Two FA test specimens are shown in Figure 5-1; the bulkhead unit's test base plate is shown.) This approach necessitated an extra assembly cycle for each unit. These tests were conducted two units at a time to the FA vibration level, 11.2 Grms for 60 seconds. The evaluation procedure was exactly the same as for the DTM qualification test.

5.2 Sticking Coefficient

No tests of the effective sticking coefficient were required on the DTM or the flight units because the assembly of the adsorbents into a structure can only affect this parameter through obscuring part of the molecular adsorber, a factor taken explicitly into account in the sizing of the units (i.e., in the presented area). However, the planned effective sticking coefficient tests of the molecular adsorber, as discussed, were not entirely successful. Fortunately, Thomson and Triolo, two interested colleagues, provided an independent verification, by a different technique, of this crucial performance parameter.³¹

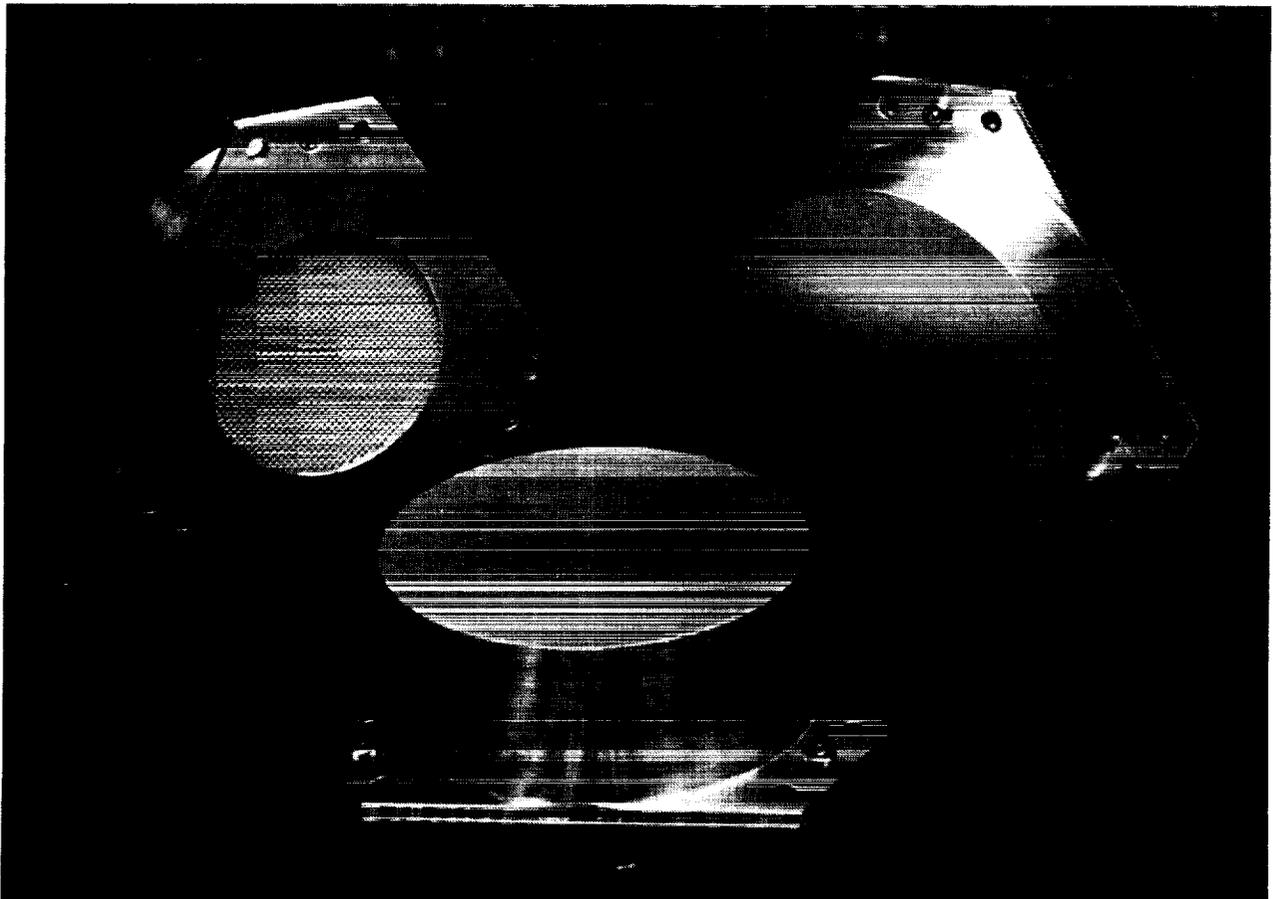


Figure 5-1. Flight acceptance vibration test arrangement. The view port for particle shedding collection is shown for the bulkhead assembly (only). Both the FA cover assembly on the left and the DTM (not shown) also have this port.

Their general approach was to incorporate a plate coated with Y preparation zeolite, supplied by JPL, into a special Goddard Space Flight Center (GSFC) test system, LAVA³², where in conjunction with the CAP analytical molecular transport model, the sticking coefficient (or molecular accommodation coefficient) of a known chemical compound could be determined through the measured effect of the adsorber on the transport of the chemical. The experimental design of Thomson and Triolo consisted of measuring the transport of palmitic acid (selected, after consultation, from the species previously studied) in the LAVA under identical conditions, first with an uncoated and then with the adsorbent-coated plate installed. The predictions of the verified CAP control model agreed with the QCM measurements, within estimated errors. The CAP model for the runs with the molecular adsorber were identical except for a sticking coefficient (of that plate) that was varied until a QCM data fit was obtained. Thomson determined the flat plate sticking coefficient to be 0.93 ± 0.01 for palmitic acid, even for the adsorber at $+55^\circ\text{C}$. In addition, Thomson used the CAP prediction of the adsorbed mass to fit the observed decrease in sticking coefficient in a linear relationship and found that the capacity of the coated plate was at least 1.1mg (corresponding to a sticking coefficient reduced to 0.2). Thomson and Triolo also found that no more than 21 percent of the adsorbed palmitic acid would desorb in 21 hours at 110°C . They also determined that the adsorber retains the cable source contamination even at 80°C .

For the estimated thickness of the zeolite coating, the sticking coefficient versus adsorbed mass result of Thomson and Triolo corresponds to a "useful" capacity of 0.12 percent by weight of adsorbent. This is much smaller than the values obtained in the present work for tetradecane and other chemicals. However, the flight adsorber assemblies together would adsorb 117mg at this capacity, which is far more than required.

Although the experimental design used by Thomson and Triolo is rather indirect for the sticking coefficient, it is a perfect simplified simulation of the action of the molecular adsorber in WFPC-2, where the geometry is much more complicated and the CCDs replace the QCMs. (See TV Test, in Section 6, below.)

This activity also provided the first attempts at coating structural flat surfaces. (See Section 7.) However, note that these stainless steel and aluminum plates were successfully coated but were not flight quality, and not tested for shedding.

5.3 Water Desorption

Water sources in the instrument such as the graphite-epoxy optical bench are known to pose an accumulation hazard on a cooled CCD if the dew point rises above the CCD temperature. For this reason, the operational plan includes a delay after launch before the CCDs are first cooled. A reasonable requirement on the total water outgassing rate from the entire complement of molecular adsorbers in WFPC-2 is that it should not further delay the start of observations, i.e., the cooling

of the CCDs. Based on the estimated conductance of the instrument and a desired dew point less than -76°C , the water desorption rate per unit area (source flux) of the adsorbers must be less than $4 \times 10^{-8} \text{ g/cm}^2\text{s}$. This value depends on the shutter being open and at least one filter in place. If the filters were also open, then $3 \times 10^{-7} \text{ g/cm}^2\text{s}$ would be allowed.

Later in the program, the water desorption of a flight adsorber (a piece of an adsorber previously removed from the instrument after the system thermal-vacuum test) was measured in a vacuum at several temperatures in MCIF No. 2. The water emission rate was monitored by periodically recording the chamber water partial pressure with an Inficon residual gas analyzer (RGA) model Quadrex 200 with an electron multiplier. Data are available for times greater than 3.5 hours, when the combined turbomolecular/ion pumps had achieved a vacuum suitable for RGA operation. The emission rate per presented area A (of the 2 cm x 3 cm x 1 in. thick piece of adsorber) Φ is related to the partial pressure of water vapor P and the system conductance for water C by:

$$\Phi = \frac{C P}{A} \quad (2)$$

Since the entire pump throat area was used to calculate the conductance, the derived values are upper limits. The results for 20°C , as controlled by a heat exchanger are given in Figure 3-3 (TV, 20C). The rate was lower than the previous result, in part, because this specimen had been stored in dry nitrogen. The more stringent required rate is reached in less than 5 hours.

After the measurements at 20°C , the temperature of the sample was increased and controlled sequentially at 45°C , 100°C , and 150°C . The pump conductance change with temperature was considered explicitly. A slight, inconsequential increase was noted at FA hot (45°C). This peak was due only to the increase in temperature; the value would have been smaller if the temperature had been 45°C from the start. Significant increases were observed only at 100°C and 150°C , and even at the higher temperature the flux decreased with time. This is the desorption of the bound water that remained after 20 hours at 20°C . These data are also given in Figure 3-4.

The final issue of water emission is whether the emission of water and other volatiles facilitates the removal of adsorbed contaminants, i.e., those species that can accumulate on a cold CCD. None of the results of these tests are diagnostic. On the basis of previous measurements of the sticking coefficient of this adsorbent, the possible loss during evacuation of the adsorber (i.e., launch) of contaminants capable of accumulating on the CCD is no more than 25 percent and probably much less. All sticking coefficient tests involved an evacuation cycle after exposure and adsorption; the adsorbents were at equilibrium with environmental relative humidity before exposure. Even if this amount of contamination were to be released by the

emission of the extremely volatile compounds present (e.g., water), and there is no evidence for this effect, it is too little to be of concern. Further, the CCDs will be too warm to collect this contamination because the time period of volatile emission is so short. Whatever is released will be pumped out through the aperture and only serve to partially regenerate the adsorbent.

Section 6

Performance in WFPC-2

6.1 System Tests

Thermal Vacuum (TV) Test

One of the objectives of the WFPC-2 system thermal vacuum test was to verify that the goal of the contamination control program for UV degradation had been met. As discussed earlier in this report, the goal could not be met without the molecular adsorber. Therefore, a sufficiently small UV loss, despite actual source rates as large as predicted, would be equivalent to an adequate performance of the flight molecular adsorber.

During the TV test the accumulation on the CCDs was measured by a proxy QCM at a different location inside the instrument. The logical connection between the QCM actual collection and the CCD predicted collection was accomplished by the use of CAP. Taylor Luan had previously performed a CAP analysis of the transport of the contaminants inside WFPC-2 with the final best estimates of the internal sources (after all bakeouts were completed), an estimate of the (flight) external rate and the molecular adsorbers in place (with a sticking coefficient of one). The QCM to CCD collection ratio also depends on the relative size of the external and internal source rates, through a dependence on source location. For this purpose, another QCM was placed outside WFPC-2 oriented to measure approximately the external flux (i.e., incident on the aperture) during the TV test. The QCM data were analyzed to separate the external flux and to infer the actual internal source rates.³³ Then the accumulation rates on the CCDs during the TV test were predicted by adjusting Luan's CAP results for these revised rates.

A measurement of the relative loss in UV throughput at 1470 Å was attempted after 48 hours of hot orbital conditions and 164 hours of nominal orbital conditions. A calibrated UV source was measured by the CCDs, which had been operating at -76°C. The predicted accumulated contamination was 50 to 100 ng/cm², which corresponds to an expected loss of 1 to 2 percent. Then the CCDs were heated to +20°C for sufficiently long enough to desorb all of the expected contamination, as determined by actual desorption tests with the QCMs. Then the UV measurement was repeated. The results were negative; no change beyond the 2 to 3 percent sensitivity of the test was observed. The molecular adsorbers clearly functioned properly.³³

After the TV test one of the flight adsorbers was removed and replaced with a spare in order to measure its condition due to the events from assembly through the TV test. The most important result was obtained by Gary Plett,³⁴ who desorbed one piece of the adsorbent at 230°C into a liquid nitrogen trap and later

measured the gas released at room temperature with a gas chromatograph/mass spectrometer (Finnigan Incos XC). He identified contaminants in WFPC-2 for the first time; benzene and octene. From his mass values and the known size of the specimen, the adsorption was only about 175 ng/cm² (mass per presented area). Because this value is far less than the expected capacity, 10 mg/cm², the adsorbers had not been ruined. Furthermore, the value from Plett's work agrees with a prediction of the collection expected during the entire TV test. Based on the modeling and testing used for the UV test³³, that value is at least 157 ng/cm². (There is no model for the hot soak test phase so the hot orbit model, which underestimates, was used.) Again, it is clear that the molecular adsorber was operating satisfactorily.

6.2 Expected in Flight

The modeling used to support the TV test was extended to separate the more and less volatile components in order to allow a valid prediction for a 30-day flight period. The nonvolatile fractions of both external and internal sources are critical for a prediction of the accumulation in 30 days because that part of the contaminant flux incident on the CCD at -76°C accumulates linearly. The separation was only approximate; the predictions range from 64 to 108 ng/cm², plus 5 ng/cm² from the flight external environment.³³ The goal was 47 ng/cm². The molecular adsorbers contributed their share to a very successful contamination reduction program, within about a factor of two of a difficult goal.

Section 7

Future Activities

The suggested work in the area of molecular adsorbers for contamination technology may be organized into the short-term future, where the goal is to improve on the present adsorber, the intermediate future, where feasibility and the utility of the effort is clear, and the long-term future, where real research holds the most ambitious, high payoff possibilities.

The short-term activities may be found simply from this account of the development of the WFPC-2 molecular adsorber. The shedding from the molecular adsorber should be reduced to virtually zero. A test program to determine the causes of shedding is needed. Even without such a program to direct a design improvement activity, the current experience suggests several design changes. Because the bulkhead units shed less than the cover units, the better design may be to clamp the retaining springs behind a shim that spans the entire lateral surface of the monolith. A method for manufacturing cordierite with more uniform thickness (especially parallel "cuts") is needed to ensure uniform clamping forces. A reduction in thickness variation among pieces would simplify the shimming operation, too. The tests that were not entirely successful should be repeated, e.g., the attempt to compare the adsorption within the first and second halves of an adsorber. In fact, this test, which could permit thinner and lighter adsorbers, should be conducted with four 0.25 in. thicknesses stacked, per the original plan. In general, in every instance when the need to produce an *acceptable* adsorber in a timely manner defeated the desire to do better, the chances for improvement are good.

In the intermediate time scale, revolutionary, not evolutionary improvements in flight adsorbers should be attempted. Zeolites tailored for efficient adsorption and high capacity for specific contaminant species should be synthesized and tested. A flat plate adsorbent coating system for (at least) aluminum should be developed and flight qualified. The promising work that had just been started on dealuminated zeolite, an adsorber that does not adsorb water, should be resumed. As part of the effort in this time scale, new tests and equipment for testing the performance of adsorbers are needed. A desorption system capable of heating and holding a sample of the adsorber at 300°C while the QCM is isolated from the thermal effects of the sample holder is required for valid sticking coefficient and capacity measurements. A new capacity test that provides a real capacity rather than a lower limit must be designed. Nothing more than an active flux monitor and a chemical reservoir, both with shutters, may be necessary. Some research with adsorbers should also be conducted. Real depth profiles in coated monoliths for known sorbates should be measured, for example.

The long-term activities are characterized by the application of this technology to the needs of aerospace contamination control, other than flight

adsorbers. There is a substantial list of such applications that have occurred to the authors during the completion of this work. Some of these applications will also be considered in the near future. As one specific, the present molecular adsorber, in combination with the development of some simple repeatable test methods, represents an almost perfect molecular contamination witness plate for use in either hardware assembly and test areas or inside vacuum chambers. With a much greater collection efficiency than a molecular fallout plate, it provides much greater overall sensitivity. Inside a vacuum chamber, it is an ideal "cold finger." It will not release its collected sample whenever there is a cryogen interruption or when the chamber is let up to ambient pressure. In fact, the sample can be simply held until the adsorber is brought to the analytical instrumentation of interest.

For the record, here is a list of the potential applications claimed for this new development:

Flight Applications

1. A flight depressurization vent for a launch contamination barrier/container for a spacecraft or instrument, or for an instrument cover to protect against the external environment during launch.
2. A spacecraft vent to protect other systems (e.g., instruments) against its outgassing during launch and flight.
3. An enhanced (suppressed reflection of contaminants) flight plume shield.

Ground Operations Applications

4. A nonflight adsorber inside an instrument to protect it during ground system testing (in effect, already done in this work with flight adsorbers).
5. An instrument contamination monitor for preflight operations (partially done in this work with flight adsorbers).
6. A passive monitor/witness plate for system thermal-vacuum tests to replace a "cold finger" (as discussed above).
7. An accelerated environmental molecular "fallout" monitor (as discussed above): enhanced collection compared to standard plate; selectivity in species by QCM temperature during desorption (reading); possible flexible substrate or removable coating to simplify reading.

8. A geometrical barrier for known sources (e.g., on GSE) in vacuum or calibration tests (also due to Taylor Luan).
9. An enhancement of the bakeout enclave when the enclave must be used in a contaminating vacuum system; no release at end of bakeout like with a cryoplate (also due to Daniel Taylor).
10. A method for measuring the accumulation on a cold surface of interest by a proxy that does not require cryogenics.
11. A collection technique for sensitive total mass loss (TML) measurements for materials with small outgassing.
12. A "sample and hold" technique for analysis of contaminants remote to the source collection site or for later redeposition for effect measurements at a remote site.
13. A method for mapping contaminant flux in a test system such as MCIF (weakly demonstrated in this work).

Research Applications

14. A controlled (by temperature) rate source of known chemical compound (preadsorbed) for contamination studies in vacuum.
15. Temperature-differential desorption from zeolite as method for chemical species separation prior to mass spectroscopy; alternative to or possible improvement over gas chromatography/mass spectroscopy (also due to P. K. Sharma).
16. A test bed for the study of the relationship between flux and sticking coefficient.

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Notes

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8. R. M. Barrer, Hydrothermal Chemistry of Zeolites, Academic Press, 1982.
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10. Alain Carpenter of Section 355 assessed this approach.
11. However, only round cross-section honeycombs are stock items. The piece in Figure 2 that is not round has been cut from a round piece. Note the missing skin or wall at the cut.
12. Faujasite is the usual name for a natural zeolite. 13X was originally a Linde designation for the synthetic version, but is now a generic designation. Unless explicitly excepted, the terms zeolite, faujasite and 13X will be used interchangeably.
13. P. K. Sharma, internal communication, December 5, 1991. The TGA measurements were conducted by Greg Hickey of Section 355.

14. D. M. Taylor, D. Soules, and D. Osborn, "The JPL Molecular Contamination Investigation Facility," in Optical System Contamination: Effects, Measurement, Control II, SPIE Proceedings Vol. 1329, ed. A. Peter Glassford, July 1990, pp. 233-244.
15. P. K. Sharma of Section 355 performed this work.
16. The optical analysis was performed by Paul Staszak, formerly of Section 355.
17. AL denotes that the cordierite surface was acid leached before any subsequent processing step.
18. This Y zeolite was synthesized per: Inorganic Synthesis, Vol. 22, edited by S. L. Holt, Jr., John Wiley & Sons, Inc., 1983, p. 64-65. For further details of the coatings, contact only G. Voecks or S. Moore.
19. The Y synthesis of the previous note.
20. Shaun R. Thomson, personal communication, EER-242-92-001/SRT, October 5, 1992. Thomson of EER Systems performed this work under contract to GSFC.
21. The design of the molecular adsorber mounting hardware was executed primarily by David Brent (formerly of Section 354) and Albany Lee (Section 358).
22. The production of these special pieces at Corning was expedited by Carol Ferratella of Corning.
23. The concept and design of these springs and the analysis of the force versus displacement rule for them are due to R. Bamford, J. Hendrickson (Section 354) and D. Brent. D. Brent also performed the analysis of the required clamping force and the stress analysis for the design.
24. J. Barengoltz, JPL internal communication, April 15, 1992.
25. The obscuration ratio, the fraction of a surface covered by particles, is an upper limit to the loss of transmission (opaque particles with no pile-up). It was chosen to characterize particle cleanliness for WFPC-2 because it is additive and permits such comparisons, and relates directly to this one optical effect.
26. J. Barengoltz, JPL internal communication, April 27, 1992.
27. Contamination Control Procedure for the Tape Lift Sampling of Surfaces, GSFC-TLS-PR-7324-1. The tape specified is Scotch 3M #480.
28. Paul Staszak, formerly of Section 355, contributed to the plan for scanning the particles on the tape, the statistical treatment, and expertly performed the scans.

29. J. Barengoltz, JPL internal communication, June 15, 1992.
30. Actually one adsorber from each batch of four.
31. Shaun R. Thomson and Jack Triolo of EER Systems, personal communication, 1992 and 1993. Triolo was formerly at GSFC for many years. This work was done under contract to GSFC. It is also an example of important work that the authors of this report hope will be published. Except as explicitly noted, the results quoted in this section are the sole work of Thomson and Triolo.
32. Chris Lorentson (GSFC), "LAVA Experimental Study CAP and SPACEII Contamination Programs," unpublished communication, June 24, 1991. The Laboratory Verification Apparatus (LAVA) was designed by Bonnie Teti (GSFC), whose work with the LAVA in verifying and comparing the CAP and SPACEII analytical molecular transport models was reported herein.
33. J. Barengoltz, internal communication, August 10, 1993.
34. Gary Plett of Section 355, internal communication, July 29, 1993. This memo in its entirety is attached to: J. Barengoltz, internal communication, August 10, 1993.



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16. Abstract <p>A device has been developed at the Jet Propulsion Laboratory, California Institute of Technology, for the adsorption of contaminants inside a space instrument during flight. The molecular adsorber was developed for use on the Wide Field Planetary Camera II, and it has been shown to perform at its design specifications in the WFPC-2. The basic principle of the molecular adsorber is a zeolite-coated ceramic honeycomb. The arrangement is efficient for adsorption and also provides the needed rigidity to retain the special zeolite coating during the launch vibrational environment. The adsorber, in other forms, is expected to be useful for all flight instruments sensitive to internal sources of contamination. Typically, some internal contamination is unavoidable. A common design solution is to increase the venting to the exterior. However, for truly sensitive instruments, the external contamination environment is more severe. The molecular adsorber acts as a one-way vent to solve this problem. Continued development is planned for this device.</p>					
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